UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2009/2010

November 2009

EBB 511/3 - Materials Characterisation Techniques

Duration : 3 hours

Please ensure that this examination paper contains FIVE printed pages before you begin the examination.

This paper consists of SEVEN questions.

Instruction: Answer **FIVE** questions. If candidate answers more than five questions only the first five questions answered in the answer script would be examined.

The answers to all questions must start on a new page.

All questions must be answered in English.

1. [a] Give an expression that describes the structure factor, F_{hkl}, for a crystal with N atoms at positions u_i, v_i, w_i. (This involves a sum of complex exponentials). For an FCC structure show that this expression yields 4f for hkl unmixed and 0 for hkl mixed.

(50 marks)

[b] An XRD analysis was carried out on chromium sample and the results are shown in the table given below:

Peak#	1	2	3	4	5	6
2θ	44.41	64.59	81.77	98.26	115.34	135.47

Determine the crystal structure using the Analytical Method. Radiation is from Cu Kα and λ = 154056 nm.

(50 marks)

2. [a] What signals are generated from a solid sample by an incident beam of high energy electrons. Identify which signals are used in SEM and which are used in TEM.

(30 marks)

[b] Are secondary electrons or backscattered electrons to be preferred for the imaging and analysing variations in the local chemical distribution on a polished sample containing aluminium and gold. Give your reasons.

(30 marks)

[c] What are the factors affecting aberration of lenses in SEM and what are the measures to be taken to minimise them?

(40 marks)

3. [a] Explain the basic principle of IR spectroscopy. What is the difference between IR and FTIR?

(30 marks)

[b] Write briefly on three applications of FTIR.

(30 marks)

[c] How are characteristic x-rays produced? How are these radiation used in Energy Dispersive Spectrometry (EDS) and discuss this technique in SEM.

(40 marks)

4. [a] Atomic Force Microscopy (AFM) is a very versatile technique for measuring surface topography. Describe the priciple and operation of an AFM. What is the advantage(s) and disadvatage(s) of non-contact mode.

(50 marks)

[b] Explain the imaging mode in TEM. Describe the difference between bright field and dark field TEM imaging modes.

(50 marks)

5. [a] Briefly describe using a schematic diagram the working of a Scanning Tunneling Microscope (STM). The answer should include the underlying principle and imaging process. What form of samples can be analysed by STM and what is its limitation.

(50 marks)

[b] In TEM imaging there are 3 primary contrast mechanisms, one, two or all three of which may contribute strongly to the appearance of a TEM image. State the mechanisms and discuss any one (1) of them. Sketch appropriate diagram(s) where necessary.

(50 marks)

6. [a] Schematically illustrate the method employed to measure the inflection temperature in a TG plot.

(20 marks)

[b] Assume that you are studying the endothermic chemical decomposition reaction of a hypothetical pure metal carbonate $MCO_3 \rightarrow MO + CO_2$ at a temperature T. You are conducting the experiment both in ambient atmosphere as well as in flowing Argon medium. Schematically illustrate and compare the possible TG-DTA plots that you may obtain.

(30 marks)

[c] Explain in brief the basic requirements that must be fulfilled while designing the furnace for a TG-DTA set up.

(30 marks)

[d] What are the effects of heating rate and sample mass on the results obtained from a TG-DTA experiment?

(20 marks)

7. [a] Consider the reduction reaction of a liquid slag CaO-20%SiO_2-40%FeO by carbon supplied through a carbon saturated liquid iron bath isothermally at three different temperatures of 1400, 1425, and 1450^{o}C. Assume that the reaction follows a first order deceleratory reaction rate model. Also assume that the change in weight of the sample is only through the evolution of carbon monoxide (CO). Illustrate the steps involved to calculate the reaction rate and the activation energy by employing the differential method of kinetic analysis.

(50 marks)

[b] Outline the mechanism of a LASER dilatometer and mention its advantages over a conventional dilatometer.

(25 marks)

[c] How does thermal conductivity of the sample and crucible affect the accuracy of the results of a TG-DTA experiment? Outline the precautions to be adopted to improve the accuracy of the results.

(25 marks)

- oooOooo -